AD-A145 051 METHOD OF OBTAINING POLYCHALCONES(U) FOREIGN TECHNOLOGY 1/1
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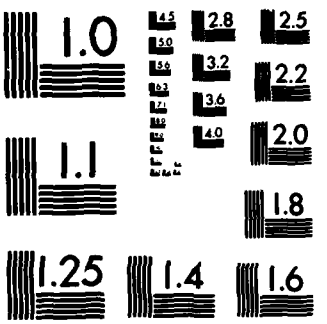

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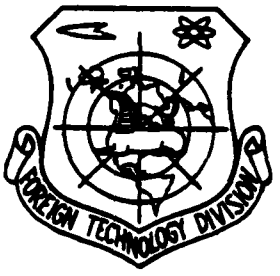
FOREIGN TECHNOLOGY DIVISION

METHOD OF OBTAINING POLYCHALCONES

by

S. V. Tsukerman, E. V. Danil'chenko and V. F. Lavrushin



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EDITED TRANSLATION

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METHOD OF OBTAINING POLYCHALCONES

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U. S. BOARD ON GEOGRAPHIC NAMES TRANSLITERATION SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	*А а*	A, a	Р р	*Р р*	R, r
Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

*ye initially, after vowels, and after ъ, ь; e elsewhere.
When written as ё in Russian, transliterate as yё or ё.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	$\sinh^{-1}$
cos	cos	ch	cosh	arc ch	$\cosh^{-1}$
tg	tan	th	tanh	arc th	$\tanh^{-1}$
ctg	cot	cth	coth	arc cth	$\coth^{-1}$
sec	sec	sch	sech	arc sch	sech^{-1}
cosec	csc	csch	csch	arc csch	csch^{-1}

Russian	English
rot	curl
lg	log

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METHOD OF OBTAINING POLYCHALCONES

Author's certificate number 376402.
Authors of invention: S. V. Tsukerman, E. V. Danil'chenko and V. F. Lavrushin

The invention concerns the obtaining of polychalcones - polymer compounds which contain in the monomer link of the macromolecule aromatic or heterocyclic nuclei, a carbonyl group, and in the α, β -position in respect to it an aliphatic double bond.

A method is known of obtaining polychalcone by means of polycondensation of aliphatic-aromatic diketone or acetone with aromatic dialdehyde with boiling in a solution of Tetralin while using as the condensing agent a 25% alcohol solution of sodium or potassium hydroxide, filtration of the reaction product, and washing it with acetone and a large quantity of water. The target product obtained in this case is very heterogeneous and a number of the chemical links of the macromolecule of the polymer contain an unsaturated grouping of carbon atoms with a hydroxyl group.

The purpose of the invention is the obtaining of polychalcones and their analogs, containing in the monomer link not only aromatic, but also heterocyclic nuclei, with a high degree of uniformity, and which are the products primarily of crotonic polycondensation.

This goal is achieved due to the fact that aromatic or heterocyclic dialdehydes are added to the reaction of polycondensation with the analogous diketones in a medium of a high-boiling solvent, diethylene glycol for example, in the presence of a solution of a strong base with heating up to 240-250°C with separation of the product of the interaction by

dilution of the mixture of the reacting substances with water, filtering, washing with hot water and alcohol, and final crotonization of the polymer by means of boiling of the dry product in acetic anhydride with subsequent purification by conventional methods.

Example 1. Obtaining of aromatic polychalcone.

1.62 g of 1,4-diacetylbenzole and 1.34 g of terephthalic aldehyde are dissolved with mild heating in 30 ml of diethylene glycol, then 5 ml of a 25% solution of potassium hydroxide is added. The mixture of the reacting substances is held at the boiling point (240-250°C) for 4-5 hours. After cooling 100 ml of water is added to the mass which is formed while mixing and the precipitated residue is filtered, washed with boiling water and hot ethyl alcohol, and dried at 100-110°C. Around 2.75 g of amorphous finely dispersed powder of a brown color is obtained.

The polycondensation product is boiled in 25 ml of freshly distilled acetic anhydride for several hours and filtered without cooling. The residue obtained is washed with alcohol and water and dried at 100-110°C. A dark-brown finely dispersed powder is obtained, forming with concentrated sulfuric acid an intensive red coloration of a halochrome nature.

The polymer obtained is not soluble in standard organic solvents, pyridine, dimethylformamide, etc., and does not melt with heating up to 500°C. The yield is 2.0-2.1 g, around 75% of theoretical.

Example 2. Obtaining of thiophene polychalcone.

1.40 g of 2,5-diformylthiophene and 1.68 g of 2,5-diacetylthiophene are dissolved with mild heating in 30 ml of diethylene glycol, then 5 ml of a 25% solution of potassium hydroxide is added. The mixture of the reacting substances is held at the boiling point (240-250°C) for 4-5 hours. After cooling 100 ml of water is added to the mass which is formed with mixing, and the precipitated residue is filtered, washed with boiling water and hot ethyl alcohol, then dried at 100-110°C. Around 2.8 g of a brown finely dispersed powder is obtained.

The polycondensation product is boiled in 25 ml of freshly distilled acetic anhydride for several hours and filtered without cooling, the residue obtained is washed with alcohol and water and dried at 100-110°C. A dark-brown powder is obtained, which with concentrated sulfuric acid

forms a green coloration of a halochrome nature. The polymer obtained is not soluble in standard organic solvents, pyridine, dimethylformamide, etc., and does not melt with heating up to 500°C. The yield is 1.8-2.0 g - around 70% of theoretical.

The resulting products possess high thermostability, electrical conductivity of the semiconductor type and paramagnetic properties.

Subject of the invention

1. A method of obtaining polychalcones by means of the reaction of polycondensation of dialdehydes and diketones in a medium of a solvent with heating in the presence of a solution of a strong base, characterized by the fact that for the purpose of obtaining products with a high degree of uniformity the reaction is conducted at 240-250°C and the intermediate products of the reaction are subjected to dehydration by boiling with acetic anhydride.

2. The method in point 1, characterized by the fact that aliphatic--aromatic or heterocyclic dialdehydes and diketones are used as the initial products.

3. The method in points 1 and 2, characterized by the fact that diethylene glycol is used as the solvent.

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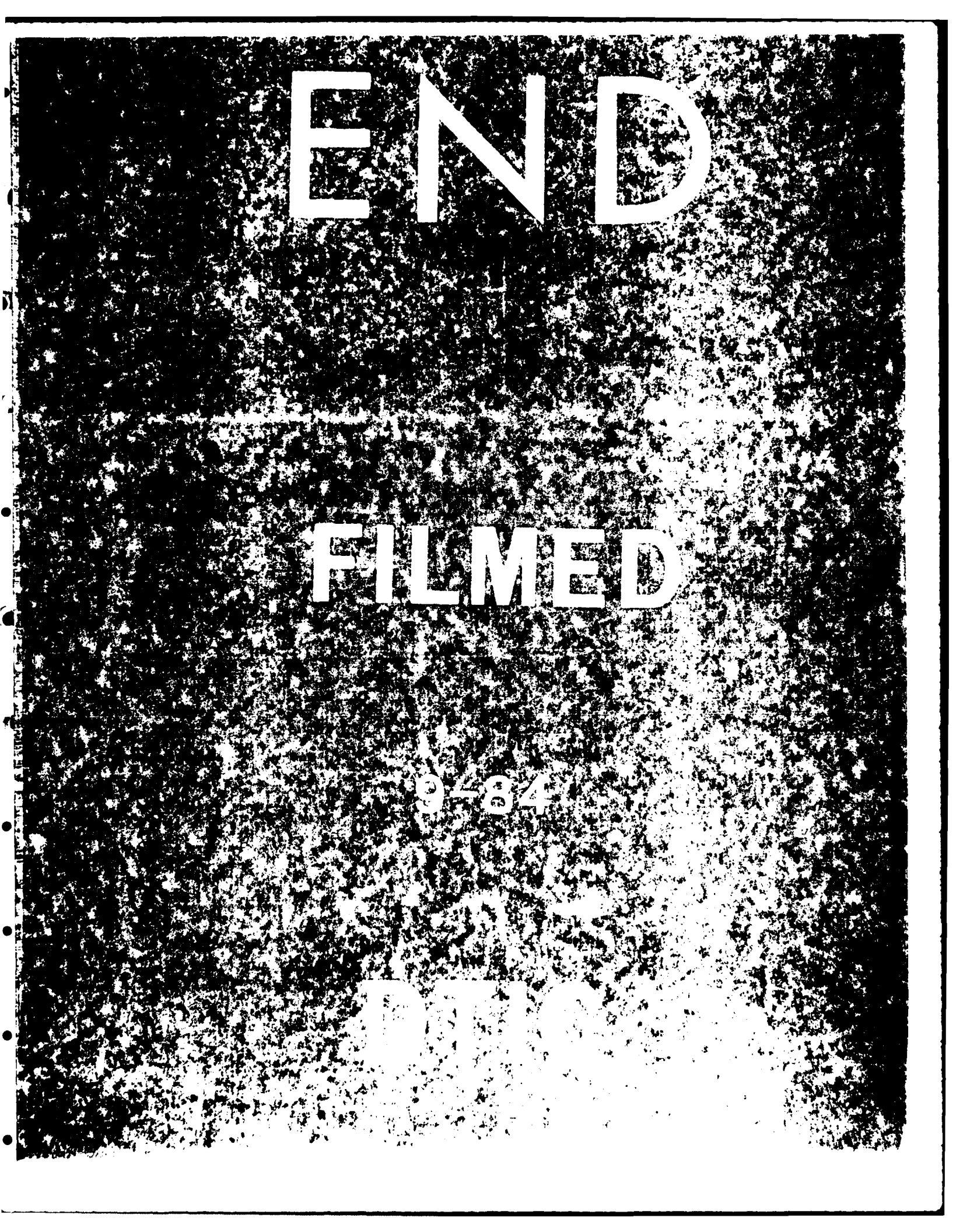
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